

**INVESTIGATION of THERMAL PROCESSING on the  
PROPERTIES of PS304, a SOLID LUBRICANT COATING**

**Summary of Research Report**

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**6/15/00 – 6/14/01**

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**Grant # NAG3-2469**

The effect of thermal processing on PS304, a solid lubricant coating, was investigated. PS304 is a plasma sprayed solid lubricant consisting of 10% Ag and 10% BaF<sub>2</sub> and CaF<sub>2</sub> in a eutectic mixture for low and high temperature lubricity respectively. In addition, PS304 contains 20% Cr<sub>2</sub>O<sub>3</sub> for increased hardness and 60% NiCr which acts as a binder. All percents are in terms of weight not volume. Previous research on thermal processing (NAG3-2245) of PS304 revealed that substrate affected both the pre and post anneal hardness of the plasma spray coating. The objective of this grant was to both quantify this effect and determine whether the root cause was an artifact of the substrate or an actual difference in hardness due to interaction between the substrate and the coating. In addition to clarifying past research developments new data was sought in terms of coating growth due to annealing.

The current research project has shown that the apparent post anneal hardness is actually reflecting to a certain degree the hardness of the underlying substrate in the as sprayed condition. Samples spray coated with varying thickness from 0.05" in to 0.20" were hardness tested in this round of research. The thicker the coating the less substrate affect was seen in both the pre and post anneal samples. The hardness appears to approach an equilibrium value in the pre-anneal condition independent of substrate, fig. 1. The results in the post-anneal condition are not quite as clear with apparent coating hardness increasing with increasing thickness on the SS304 while doing the opposite on X750, fig. 1. This anomalous behavior may be due to the coating growth described below. Post-anneal we are probably seeing a combination of the coating hardness, the substrate hardness and the growth layer hardness. This will be investigated utilizing a micro-hardness tester in the next round of testing. All current hardness measurements were made on the Rockwell B scale.

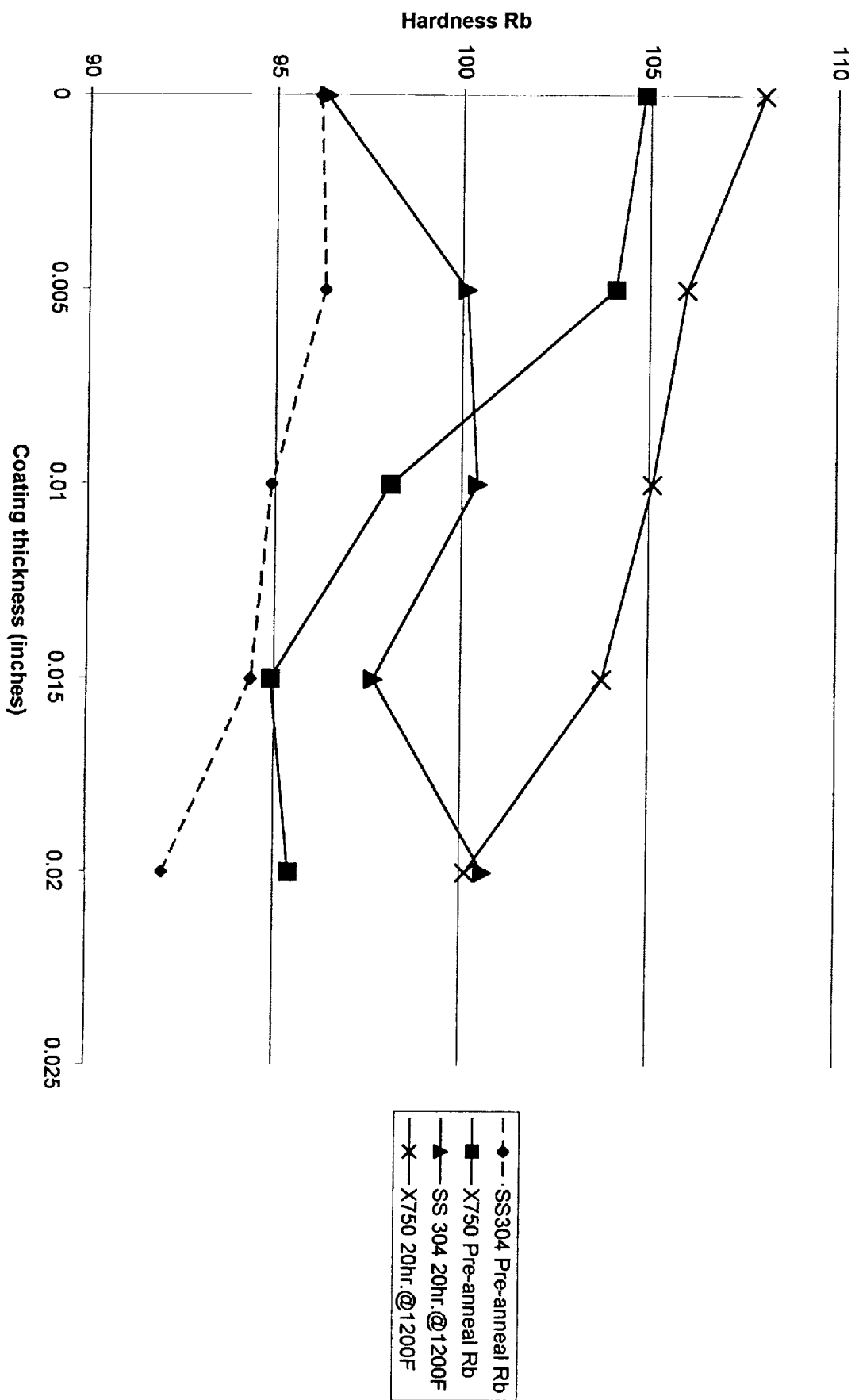
Coating growth was found to be a function of substrate, time at temperature and coating thickness, figs 2 and 3. It is postulated that a diffusion reaction mechanism is occurring. On a substrate of Inconel X750 as the coating thickness increases the % coating growth decreases for all thermal processes. On Stainless 304 substrates the % coating growth is increasing approximately linearly with respect to coating thickness until the annealing temperature is raised to 1200F. At 1200F the coating growth initially increases with coating thickness then falls off as the thickness is increased further. It would appear that there is a minimum temperature for reactant (Oxygen?) diffusion and a maximum diffusion depth based on time at temperature. Additional investigation into these phenomena will also be done during the next round of testing.

In addition to the previously described results thermal processing effects on adhesion were also investigated. Results of this research are contained in NASA Tech Memo 2001-210944. The abstract of this report is attached.

The hardness and coating growth data are being consolidated into a paper that will be submitted for publication in Tribology Transactions and presentation at the STLE Annual Meeting in 2002.

No inventions were developed under the auspices of this research grant.

Fig. 1 Rb Hardness as a function of coating thickness



**Fig.2 Coating growth on Stainless 304**

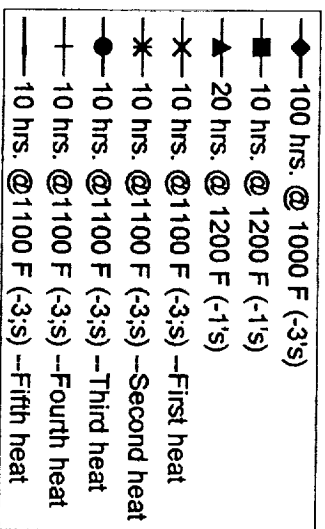
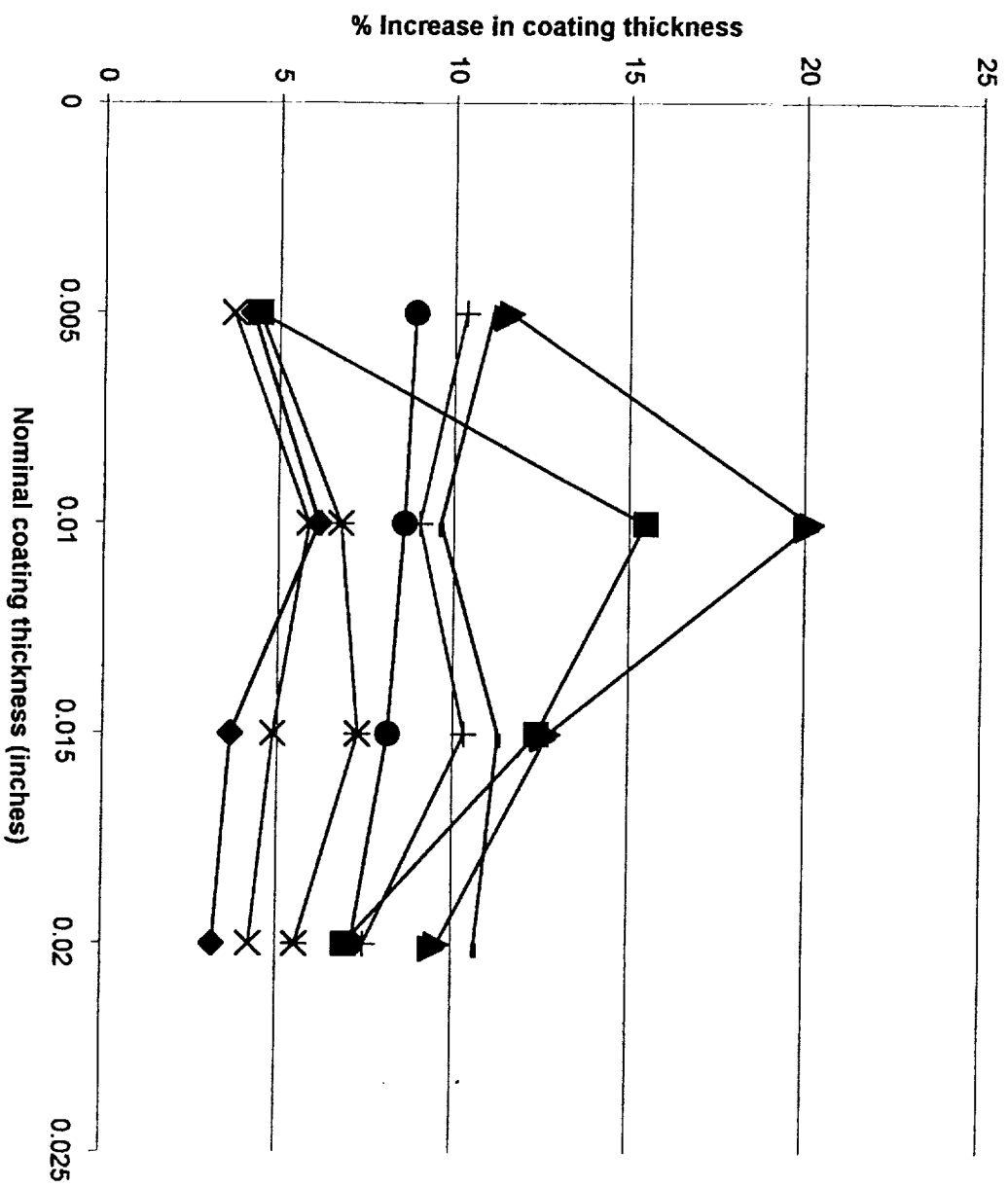
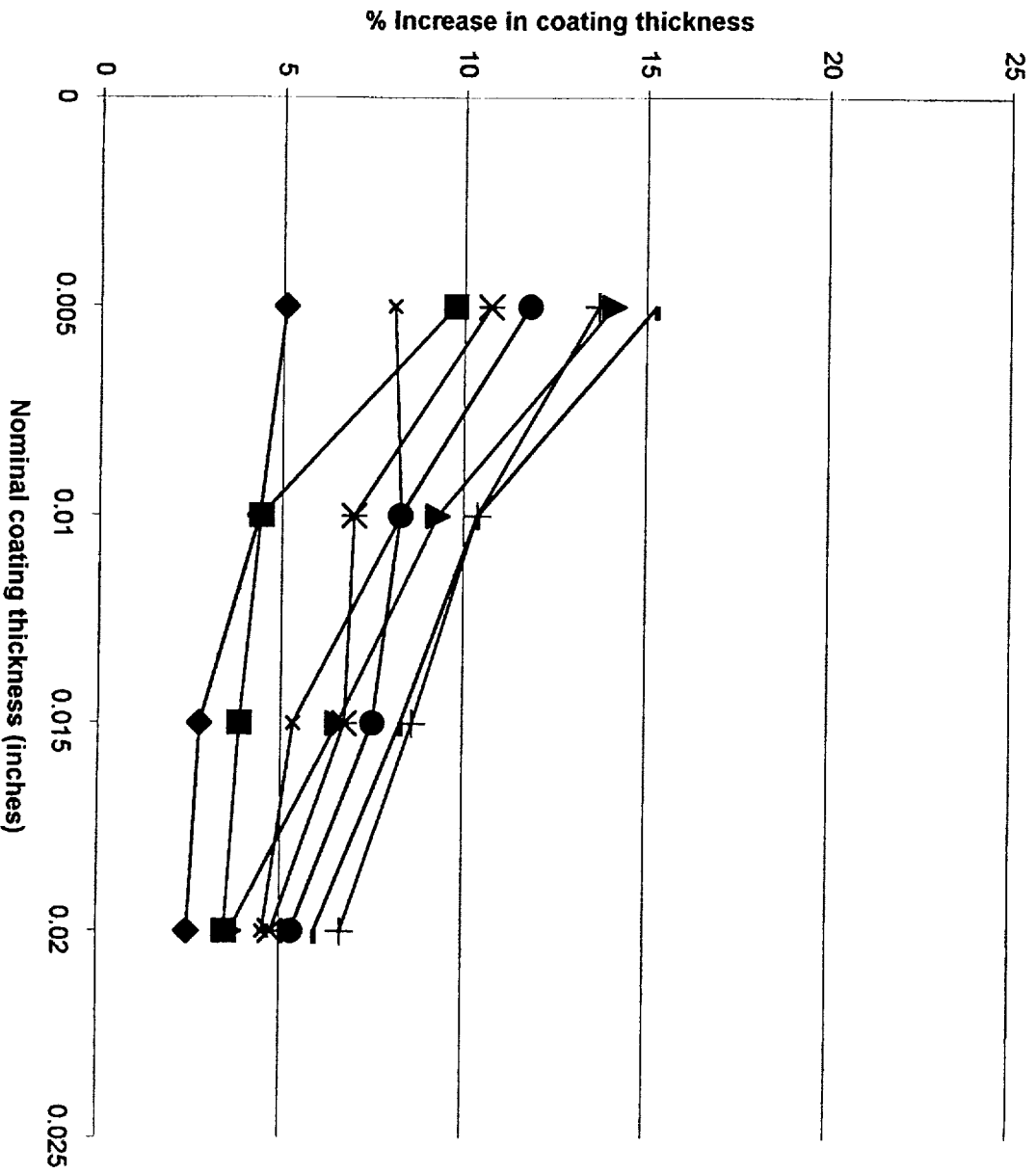


Fig. 3 Coating growth on x750



- ◆ 100 hrs. @ 1000 F (-3's)
- 10 hrs. @ 1200 F (-1's)
- ▲ 20 hrs. @ 1200 F (-1's)
- ✱ 10 hrs. @ 1100 F (-3;s) --First heat
- ✱ 10 hrs. @ 1100 F (-3;s) --Second heat
- 10 hrs. @ 1100 F (-3;s) --Third heat
- + 10 hrs. @ 1100 F (-3;s) --Fourth heat
- 10 hrs. @ 1100 F (-3;s) --Fifth heat

| REPORT DOCUMENTATION PAGE   |   |  | Form Approved<br>OMB No. 0704-0188   |  |
|---|---|--|--|--|
| Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.  |   |  |  |  |
| 1. AGENCY USE ONLY (Leave blank)  |   | 2. REPORT DATE<br>May 2001                                 |  | 3. REPORT TYPE AND DATES COVERED<br>Technical Memorandum |
| 4. TITLE AND SUBTITLE<br><br>Thermal Processing Effects on the Adhesive Strength of PS304 High Temperature Solid Lubricant Coatings   |   |  | 5. FUNDING NUMBERS<br><br>WU-708-18-13-00                                    |  |
| 6. AUTHOR(S)<br><br>Christopher DellaCorte, Brian J. Edmonds, and Patricia A. Benoy   |   |  |  |  |
| 7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)<br><br>National Aeronautics and Space Administration<br>John H. Glenn Research Center at Lewis Field<br>Cleveland, Ohio 44135-3191   |   |  | 8. PERFORMING ORGANIZATION<br>REPORT NUMBER<br><br>E-12798                   |  |
| 9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)<br><br>National Aeronautics and Space Administration<br>Washington, DC 20546-0001   |   |  | 10. SPONSORING/MONITORING<br>AGENCY REPORT NUMBER<br><br>NASA TM-2001-210944 |  |
| 11. SUPPLEMENTARY NOTES<br><br>Christopher DellaCorte and Brian J. Edmonds, NASA Glenn Research Center; and Patricia A. Benoy, St. Louis University, St. Louis, Missouri 63103. Responsible person, Christopher DellaCorte, organization code 5960, 216-433-6056.   |   |  |  |  |
| 12a. DISTRIBUTION/AVAILABILITY STATEMENT<br><br>Unclassified - Unlimited<br>Subject Category: 23<br><br>Available electronically at <a href="http://gltrs.grc.nasa.gov/GLTRS">http://gltrs.grc.nasa.gov/GLTRS</a><br>This publication is available from the NASA Center for Aerospace Information, 301-621-0390.  |   |  | 12b. DISTRIBUTION CODE   |  |
| 13. ABSTRACT (Maximum 200 words)<br><br>In this paper the effects of post deposition heat treatments on the cohesive and adhesive strength properties of PS304, a plasma sprayed nickel-chrome based, high temperature solid lubricant coating deposited on stainless steel, are studied. Plasma spray deposited coating samples were exposed in air at temperatures from 432 to 650 °C for up to 500 hr to promote residual stress relief, enhance particle to particle bonding and increase coating to substrate bond strength. Coating pull off strength was measured using a commercial adhesion tester that utilizes 13 mm diameter aluminum pull studs attached to the coating surface with epoxy. Pull off force was automatically recorded and converted to coating pull off strength. As deposited coating samples were also tested as a baseline. The as-deposited (untreated) samples either delaminated at the coating-substrate interface or failed internally (cohesive failure) at about 17 MPa. Samples heat treated at temperatures above 540 °C for 100 hr or at 600 °C or above for more than 24 hr exhibited strengths above 31 MPa, nearly a two fold increase. Coating failure occurred inside the body of the coating (cohesive failure) for nearly all of the heat-treated samples and only occasionally at the coating substrate interface (adhesive failure). Metallographic analyses of heat-treated coatings indicate that the Nickel-Chromium binder in the PS304 appears to have segregated into two phases, a high nickel matrix phase and a high chromium precipitated phase. Analysis of the precipitates indicates the presence of silicon, a constituent of a flow enhancing additive in the commercial NiCr powder. The exact nature and structure of the precipitate phase is not known. This microstructural change is believed to be partially responsible for the coating strength increase. Diffusion bonding between particles may also be playing a role. Increasing the heat treatment temperature, exposure time or both accelerate the heat treatment process. Preliminary measurements indicate that the heat treatment also results in a one time, permanent coating thickness increase of about 3 percent. Based upon these results, the incorporation of a heat treatment prior to final finishing has been incorporated in the application process of this coating technology. |   |  |  |  |
| 14. SUBJECT TERMS<br><br>Coatings; Lubrication; Solid lubricants  |   |  | 15. NUMBER OF PAGES<br>17  |  |
|   |   |  | 16. PRICE CODE   |  |
| 17. SECURITY CLASSIFICATION<br>OF REPORT<br>Unclassified  | 18. SECURITY CLASSIFICATION<br>OF THIS PAGE<br>Unclassified | 19. SECURITY CLASSIFICATION<br>OF ABSTRACT<br>Unclassified | 20. LIMITATION OF ABSTRACT   |  |

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|--|--------------------|--|
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and Address

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NASA Grant Title:

Investigation of Thermal Processing on the properties of  
PS 304, a SOLID LUBRICANT COATING

NASA Grant Number:

NAG 3-2469

NASA Grant Monitor:

Syretta Williams

Grant Completion Date:

6/14/01

Today's Date:

1/15/02

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2. Size of Business: ( ) Small ( ) Large (X) Nonprofit Organization
3. Have any nonpatentable new technology items resulted from work performed under this grant during this reporting period? ( ) yes (X) no
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5. Are new technology items (nonpatentable or patentable) being disclosed with this report?  
( ) yes (X) no

## II. New Technology Items

Please provide the title(s) of all new and previously disclosed new technology items conceived or first reduced to practice under this grant.

| Title    | Internal Docket<br>Number | Patent Appl.<br>Filed | Patentable<br>Item | Nonpatentable<br>Item |
|----------|---------------------------|-----------------------|--------------------|-----------------------|
| 1. _____ | _____                     | ( )                   | ( )                | ( )                   |
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Patricia A. Benoy Associate Prof. (PATRICIA A. BENOY)  
Name and Title of Authorized Official

Patricia A. Benoy 1/14/02  
Signature and Date

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